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J.L. Jambor
Leslie Research and Consulting

J.E. Dutrizac
Mineralogy and Metallurgical Processing Program

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THE EFFECT OF PARTICLE SIZE ON THE NEUTRALIZATION POTENTIAL IN STATIC-TEST PREDICTIONS OF ACID DRAINAGE

John Leslie Jambor[1] and John E. Dutrizac[2]

[1]Leslie Research and Consulting, 316 Rosehill Wynd
Tsawwassen, British Columbia, Canada V4M 3L9
e-mail: jljambor@aol.com
[2]CANMET, 555 Booth Street, Ottawa, Ontario, Canada K1A 0G1
e-mail:jdutriza@nrcan.gc.ca

Abstract

Thousands of static tests are performed annually to predict whether mine wastes and overburden have the potential to yield acid rock drainage (ARD); that is, whether the acid-generating potential (AP) is greater than the neutralization potential (NP). One of the factors that affects the rate of acid neutralization is particle size. The most widely used static test, commonly referred to as the Sobek test, specifies that the particle size be minus 60 mesh (<250 μm), but other static test methods for the same prediction of potential ARD specify different particle sizes. None of the procedures requires that the surface area of the sample be measured. To understand the effect that particle size has on the neutralization potential, monomineralic samples of various minerals have been tested for their NP, and for their NP at different particle sizes. The NP values consistently increase as the particle size decreases for almandine garnet, grossular garnet, zoisite, amphibole and calcic plagioclase. However, the results revealed that normalization of the NP to the BET-determined unit surface area can give misleading results because the NP values are not uniformly proportional to the surface area of material that has been screened to only a single passing size. Hence, a low measured surface area for a particular mineral with a low directly-measured NP may yield an extraordinarily high normalized NP. The results further suggest that, for the comparison of the NP values contributed by minerals, it is more realistic to use the measured NP than the normalized NP. Experiments with screened fractions constrained to a restricted size range indicate that the NP values for anorthite and forsterite are strongly affected by particle size, with the NP increasing as the particle size decreases. Hence, any factor that results in increased amounts of fines in a test-sample, such as overgrinding, will also have the effect of increasing the measured NP.

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Introduction

Acidic drainage derived from the oxidation of sulfides is a worldwide phenomenon that may occur naturally but is most commonly associated with anthropogenic activities, especially the oxidation of sulfide-bearing mine wastes. The problem of acid drainage, which is commonly referred to as acid rock drainage (ARD) or acid mine drainage, arises because sulfides oxidize rapidly whereas the associated non-sulfide minerals that may be capable of attenuating the acidity are either slower to react or, upon dissolution, release insufficient base to attenuate the acidity. Thus, the prediction of whether wastes will release ARD is dependent not only on the accurate determination of their acid-producing potential (AP) and their capacity for base release (or neutralization potential, NP), but also on the extrapolation of these parameters to the rate at which natural weathering of the wastes will occur.

The method of determining the AP and NP of a sample is commonly referred to as a static test because it is a one-time determination that, in contrast to kinetic tests, yields no data on weathering rates. Nevertheless, because static tests can be performed simply, rapidly, and inexpensively, they are widely used by the coal- and metal-mining industries as a screening method to assess whether further, more elaborate tests are warranted. Although various static-test methodologies have been proposed, the most widely used is that of Sobek et al. [1]. The protocol by Sobek et al. specifies that the sample to be tested be ground to pass 60 mesh (<250 μm), but different particle sizes are used in other static-test methodologies [2, 3, 4]. None of the methodologies requires that the surface area of the test-sample be determined. It is well known, however, that the rate or extent of chemical reactions is generally affected by surface area. Static tests are simple chemical tests, and it was shown [4] that Sobek NP values of three rock samples increased as the particle size was decreased from 0.6 mm to –60, –150, and –325 mesh. Similar particle-size effects have been observed for the rates of weathering of waste rocks [5].

As part of an extensive program to test the NP contributed by specific minerals [6, 7], the influence of particle-size variations on the Sobek NP was investigated. Although particle size and grain size are commonly used interchangeably in the literature, here we use the concept that a particle is an entity that may be monomineralic or polymineralic. A particle may be monomineralic yet polycrystalline; i.e., composed of several grains of the same mineral. Particles and grains are synonymous only when the particle consists of a single crystal.

Sample Preparation

Monomineralic samples were prepared as in [6] and the compositions were determined by electron microprobe analysis [7]. Unless noted otherwise, crushing was non-mechanical, dry screening was utilized except at –325 and finer meshes, and the NP values were determined at B.C. Research Inc., Vancouver, Canada, using standard Sobek [1] procedures. The surface areas of the test samples were obtained at CANMET using 70% neon and 30% nitrogen as the adsorbate gas in the BET method.

Normalization of NP and Surface Area

Even with standardized preparation procedures, pulverized minerals screened to pass a particular mesh size are unlikely to have identical surface areas for the screened fraction. Factors such as cleavages, aspect ratios, grain sizes, and especially the variable propensity to form fines will affect the surface area of the screened fraction. Consequently, a more

representative measure of the surface exposure is the BET-determined surface area of the screened sample. For comparative purposes, it would be convenient if the NP values of various minerals were to be normalized to a unit surface area, expressed as m^2/g. Such normalization was done in the initial study of the NP of minerals [6] and seemed to give acceptable results; an exception, however, was that olivine gave an NP of 23.9 and an inordinately high normalized NP of 199 (all NP values herein are kg $CaCO_3$ equivalent per tonne of material). A similarly abnormal value in the current study was obtained for a sample of garnet even though it had been prepared no differently than the other samples.

Figure 1 shows the normalized NP versus Sobek NP for three samples. It is evident that the NP increases with decreasing particle size, but that the variation is only 2-3 units of NP despite the change from –60 to –325 mesh (–250 to –44 μm). For almandine garnet of composition alm_{49}, the NP normalized to unit surface area decreases from 98.1 for the sample screened to –60 mesh, to 21.9 for the sample screened to –325 mesh. Thus, the normalized NP has decreased enormously, by 76.2 units, whereas the measured, non-normalized NP has increased by 1.6 units. These results indicate that, in some instances, normalization of the NP values can be unduly influenced by surface area rather than having a direct relationship to NP; hence, normalization for comparison of the NP results from different minerals may result in misleading conclusions. Anbeek [8] concluded from experimental results that the dissolution rates of fresh feldspar surfaces, when normalized to the BET surface area, are valid at one particle size only; hence, the dissolution rates cannot be compared if the particle sizes are different [8].

Figure 1. Relationship between NP and the normalized NP for zoisite (epidote group) and two samples of almandine (Fe-Al garnet), each screened to –60 and –325 mesh. The NP at the finer grain sizes increases only slightly, but the normalized NP decreases substantially.

The relationship between the measured NP and particle size is further illustrated in Figure 2. Predictive techniques typically do not demand a particle size as fine as –325 mesh, and it is apparent that the NP variation is relatively small over the size ranges either in use or expected to be used if new static tests are devised. At particle sizes finer than about –200 mesh (75 μm), however, the NP values begin to increase rapidly as the particle size decreases (Figure 2b). The results therefore suggest that the proportion of fines in a sample will affect the NP value as much or more than the selection of the particular screen size typically applicable to static tests.

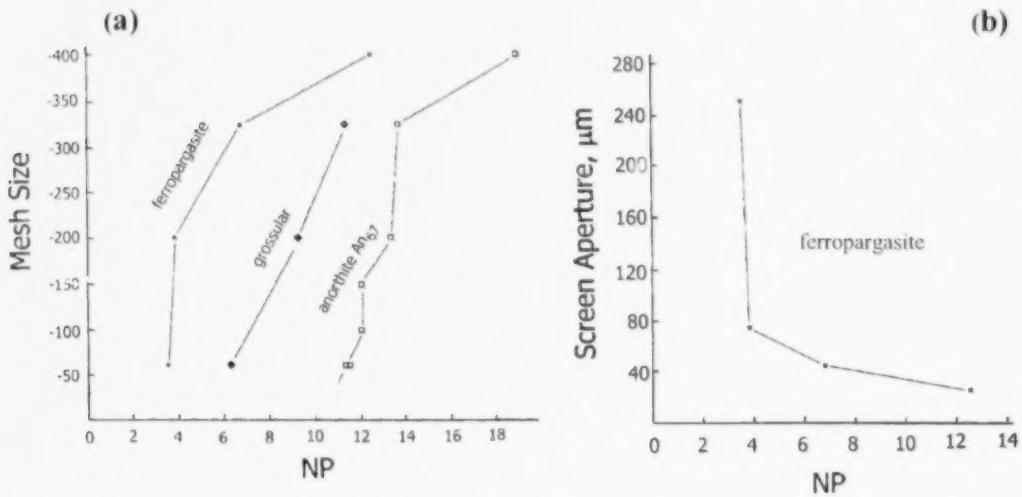

Figure 2. (a) Left diagram shows the increase in NP for ferropargasite (amphibole), grossular (Ca-Al garnet), and anorthite (calcic plagioclase feldspar) as the particle size decreases in accordance with the mesh size. (b) The same data for ferropargasite, but plotted for screen aperture rather than mesh designation.

Effect of the Method of Sample Preparation

Except for asbestiform-type minerals, which required mechanical grinding and wet screening, all samples in this study were prepared by repeated crushing of a coarse specimen with a hammer until successive screenings yielded sufficient mass for the NP tests. This preparation method likely minimized the quantity of fines relative to those obtainable by mechanical processing, which generally is less controllable in terms of "overgrinding".

An examination of the effect of the sample-preparation technique was prompted by the high NP value (29.5) obtained from a phlogopite sample in which no carbonate minerals were detectable by optical microscopy or infrared spectroscopy. Treatment of a portion of the sample with acetic acid reduced the NP, but only to 24.9. Consequently, it was speculated that, because the original specimen was a centimeters-wide single plate that had required a considerable amount of impact to reduce it to –60 mesh, the preparation procedure may have led to the development of an inordinately large number of strain sites in the mica. Strain sites and other crystallographic defects are well-known to be the loci for increased susceptibility to acid dissolution. To examine this hypothesis, the phlogopite and two other minerals

(chlorite and vermiculite) that were originally coarse, single plates, and which gave high NP values, were tested. Because sample availability was limited, for the chlorite and vermiculite it was necessary to use residual fractions that had already been hammer-crushed to 1-3 mm. For the phlogopite, however, the original specimen was about 1 cm thick; thus, a new sample was cleaved, and both the hammer-crushed 1-3 mm remnant and the new sample were tested separately. To provide a contrast, it was thought that milling of the samples in a household coffee grinder might lessen the presumed strain effects.

Table 1 shows the results from the hammer-crushed versus the milled samples. Most notable is that the new sample of phlogopite, for reasons unknown, has a much lower NP that is more in accord with the results obtained from other, unrelated samples [6]. The change in NP for the hammer-crushed versus milled vermiculite is negligible, but all other samples show a reduction in the NP for the milled samples. Thus, the method of sample preparation may not only affect the amount of fines that is generated, but may also induce physical changes, such as increased numbers of defects, that accelerate dissolution and hence increase the NP.

Table 1. NP of hammer-crushed (HC) versus milled samples

Mineral	HC	Milled
phlogopite, original	29.5	25.0
phlogopite, new	8.8	7.8
clinochlore chlorite	12.5	8.3
vermiculite-hydrobiotite	29.0	28.7

Effect of Particle Size

As has been noted, different static tests specify different screen sizes for sample preparation, and it has been shown (Figure 2) that the NP increases as the particle size decreases. Because material screened to pass a specified mesh will contain all sizes finer than that mesh, it was thought that a more precise relationship would be to compare the NP values to the BET-measured surface area of the samples. Figure 3 shows such a plot for three minerals at various screen sizes; for example, the data for grossular show the NP for a sample screened to –60 mesh, another screened to –200 mesh, and the third screened to –325 mesh. All are otherwise the same material.

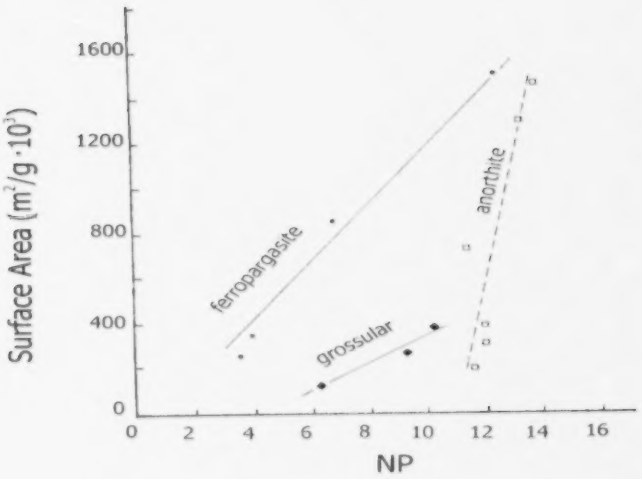

Figure 3. NP versus the BET-measured surface area for ferropargasite amphibole, grossular garnet, and anorthite plagioclase. Trend lines are approximations only, but the results if extrapolated suggest that NP for the grossular and anorthite would apparently exist even at zero surface area.

Figure 3 shows that the NP increases as the specific surface area increases, and that the relationship differs for different minerals (hence the variation in slopes). For the range of surface areas studied, it is evident that the NP is directly proportional to the surface area. However, the slopes for grossular and anorthite clearly do not extrapolate through the origin, thus leading to the impossibility that those minerals would still have NP at zero surface area.

It has been noted that surface areas measured by gas adsorption may not be an appropriate parameter to measure dissolution rates if the internal (mesopore and micropore) surfaces of a particle do not dissolve at the same rate as the external surface [9,10]. Much discussion in the literature has been concerned with weathering rates or laboratory dissolution rates versus surface area or specific crystallographically reactive sites [9-15], and it has been suggested [16] that geometric rather than BET surface areas may provide a more accurate parameter for estimating dissolution rates. Dissolution rates do not necessarily correspond to NP values, but there is a simple relationship insofar as silicates and aluminosilicates that are slow to dissolve will inevitably have low NP values.

Relationship of NP to Geometric Surface Area

Because static-test samples contain particles of all sizes finer than that of the specified screening, it would be difficult and time-consuming to derive accurately an average particle size for the screened fraction; moreover, such an average would likely be of questionable value because of the effect of fines on the NP values (Figure 3). Therefore, to gain insight into the relationship between NP and particle size, samples of anorthite (An 65, Harp Lake, Newfoundland–Labrador) and forsterite (Fo 91, San Carlos, Arizona) were screened to constrained fractions as shown in Table 2. After screening, the fines were minimized by ultrasonic agitation of the sample immersed in acetone, decanting of the cloudy supernatant

solution, and repeating the process through 4-6 cycles. Although the agitation may not have resulted in the removal of all of the fines, the cleaning was considered to be adequate relative to the variables inherent in the Sobek test.

Table 2. Particle size and NP of anorthite and forsterite

Sample No.	Screen Mesh	Aperture Mm	d_c μm	A_{geo} m^2/g	A_{BET} m^2/g	λ	NP
Anorthite							
240	–60+70	250-212	230.5	0.0096	0.136	14.2	3.7
241	–70+80	212-180	195.6	0.0113	0.167	14.8	4.2
242	–80+100	180-150	164.5	0.0134	0.157	11.7	4.7
243	–100+140	150-105	126.2	0.0175	0.184	10.5	5.7
244	–140+170	105-90	97.3	0.0227	0.196	8.6	5.5
245	–170+200	90-75	82.3	0.0269	0.200	7.4	5.0
246	–200+230	75-63	68.8	0.0321	0.220	6.9	7.0
248	–270+325	63-44	52.8	0.0421	0.229	5.4	8.5
249	cyclone	44-25	33.6	0.0658	0.247	3.8	10.0
250	cyclone	<25	7.8	0.2833	1.139	4.0	16.4
Forsterite							
261	–60+70	250-212	230.5	0.0078	0.0369	4.7	29.4
262	–80+100	180-150	164.5	0.0109	0.0537	4.9	34.8
263	–140+170	105-90	97.3	0.0185	0.0919	5.0	40.3
264	–230+270	63-53	57.9	0.0310	0.1028	3.3	42.8*
265	–270+325	53-44	48.4	0.0371	0.1159	3.1	60.2**
266	–325	<44	11.6	0.1549	0.3784	2.4	89.6

* titrant volume <3 mL but insufficient sample to repeat

** 1 g sample used

Table 2 also lists the geometric surface areas, A_{geo}, calculated [17, 18] by assuming that the particles are smooth spheres: $A_{geo} = 6/\rho d_c$, where ρ is the density (taken as 2.715 g/cm^3 for anorthite, and 3.34 g/cm^3 for forsterite), and $d_c = (d_{max}–d_{min}) / ln(d_{max}/d_{min})$, where d_{max} and d_{min} are the maximum and minimum particle sizes as indicated by the screen apertures. The computed roughness factor, λ, (Table 2) is A_{BET}/A_{geo}, and reflects the difference between the assumed smooth spherical surface and that measured by the gas-adsorption (BET) method. Both A_{geo} and A_{BET}, as expected, increase as the particle size becomes finer. The surface-roughness factor, λ, decreases with decreasing particle size. For the nine samples of anorthite that have a constrained size range, plots of λ versus A_{BET} and versus A_{geo} have different slopes that begin to diverge substantially once the particle size is finer than about 90 μm. For the forsterite, however, fewer points are available, the scatter is greater, and the relationship is obtuse.

Figure 4a shows that the BET-determined surface area of the samples increases substantially once the particle size is finer than 20-40 μm. Above this range, forsterite has a surface area lower than that of anorthite, which is in agreement with the data of Brantley and Mellott [9]. The roughness factor of forsterite is also lower (Figure 4b), indicating that the olivine has fewer topographic features and lower porosity than the plagioclase. The roughness factors for

both forsterite and anorthite should trend to a limiting finite value as the particle size decreases, as is suggested by Figure 4b.

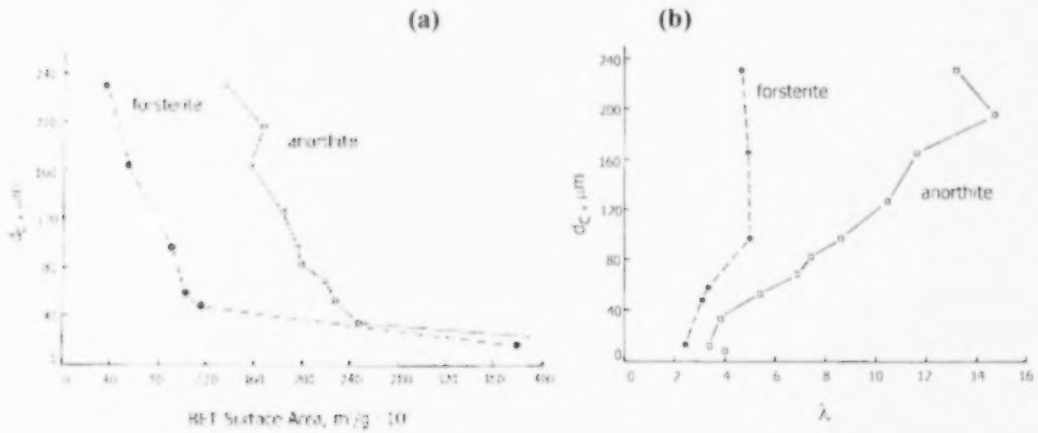

Figure 4. Average particle diameter (d_c) versus BET-determined surface area (Figure 4a) and versus the roughness factor (λ, Figure 4b) for screened fractions of forsterite and anorthite (Table 2).

For particles screened to constrained sizes, the data in Table 2 indicate that the NP increases as the particle size decreases. Figure 5 illustrates that, for both anorthite and forsterite the NP increases as the roughness factors and geometric surface areas decrease. Comparison with Figure 4 indicates that, notably for forsterite, particle size rather than roughness dominates the relationship with NP.

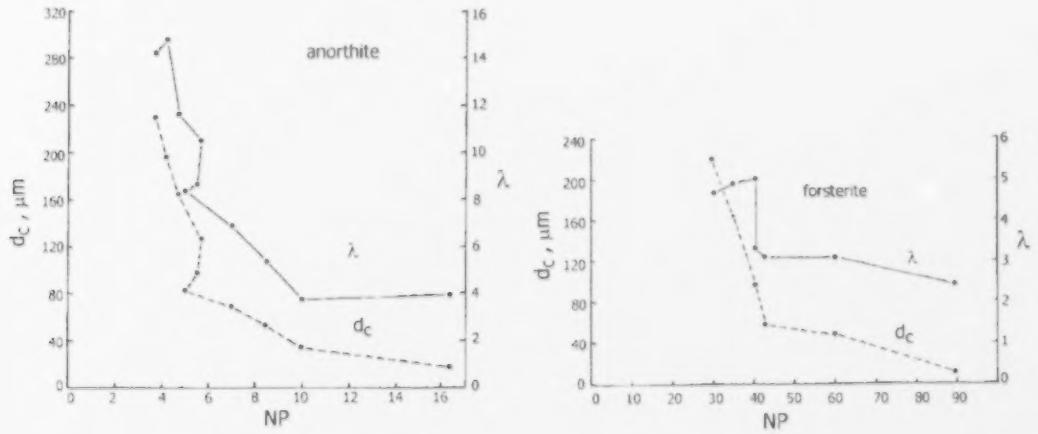

Figure 5. NP versus geometric diameter (d_c) and versus the roughness factor (λ) for anorthite and forsterite.

It was shown previously (Figure 2) that the NP changes little if samples are screened only to pass a specific mesh size but with no restriction on particle sizes within the passed fraction. Such fractions have an apparent, but not possible, relationship in which the samples would still have NP even at zero mesh size (Figure 2). If, however, the samples are screened to a specific size range (Table 2), a more reasonable relationship is obtained. For the BET-determined surface areas, Figure 6 shows not only that the NP decreases as the surface areas decrease, but that projections to zero NP at zero surface area are likely trends for both anorthite and forsterite. Figure 7 illustrates that the relationship between A_{geo} and NP also plausibly trends toward the origin; i.e., zero NP at zero A_{geo}. The important conclusion from a mineralogical and environmental perspective is the demonstration that the NP of a sample is strongly influenced by the amount of fines in the sample. For example, the NP of anorthite that passed 60 mesh is 3.7, but the NP almost doubles for the fraction that passed 200 mesh. Likewise, for the same criteria, the NP of forsterite increases by at least a third. Thus, the NP can be strongly influenced by the amount of fines generated during sample preparation. Moreover, the experimental data indicate that it is not valid to disregard the effect of particle size in comparisons of Sobek results with those of other static-test methodologies in which a finer screen-passing particle size is specified. Although the NP values of bulk, unsorted samples change by only a few units, it is evident that the effect of utilizing a passing size finer than minus 60 mesh is to increase rather than decrease the NP relative to the values obtained by the Sobek method.

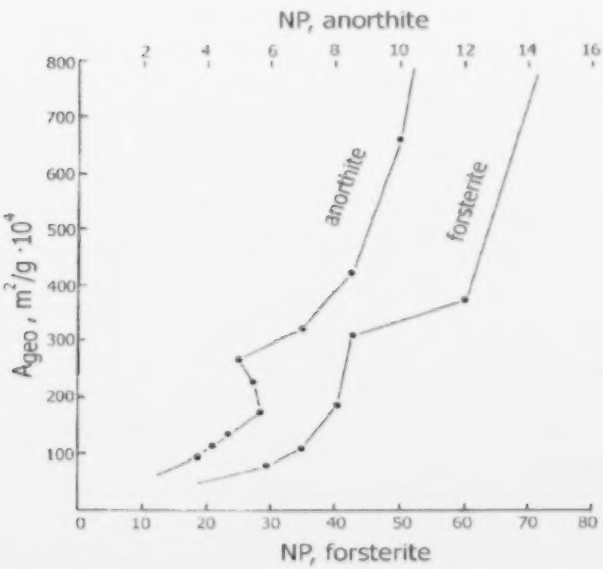

Figure 6. NP values versus BET-measured surface areas of forsterite and anorthite screened to the limited particle-size ranges given in Table 2. The trend line for anorthite at the highest surface area is a projection toward the 1,139 value for the <25 μm fraction, which is off scale.

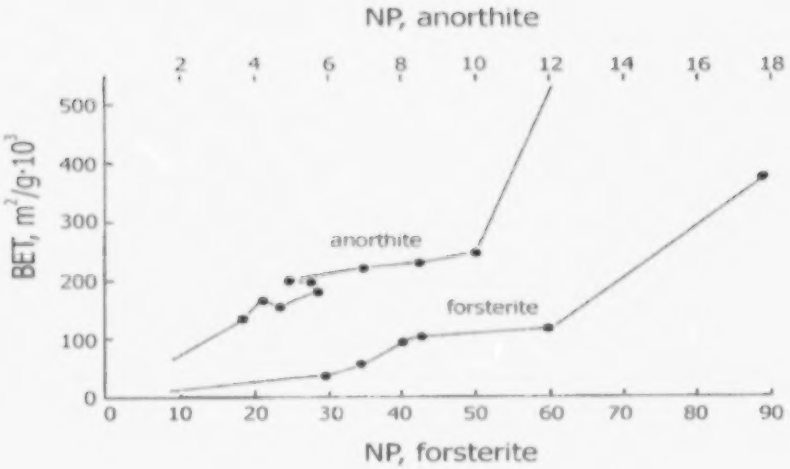

Figure 7. NP values versus geometric surface areas for anorthite and forsterite screened to the particle sizes given in Table 2. The trend line for anorthite at the highest surface area is a projection toward the off-scale value of 2,833 for the <25 μm fraction; similarly, the trend line for forsterite is toward 1,549 for the <44 μm fraction.

NP Variations for Serpentine

Sobek tests of antigorite and lizardite serpentines [6] gave NP values of 15.1 and 16.1, respectively. These results, obtained from relatively coarse-grained, display-type specimens, seemed to be at variance with the laboratory behavior of serpentine tailings obtained from the Magnola operation near Asbestos, Quebec [19]. Magnola extracts Mg from serpentine tailings (>90% serpentine) originating from the JM asbestos mine.

The Magnola tailings samples consist predominantly of lizardite and antigorite [20] and are carbonate-free as indicated by the absence of effervescence in HCl. Data for various screened fractions of the tailings are given in Table 3. The substantially higher NP (45-50) compared to the values reported previously [6] for lizardite and antigorite is partly attributable to the presence of small amounts of brucite in the tailings; however, the major difference is in the grain (sensu stricto) size. Thus, a notable feature in Table 3 is that the BET-measured surface area is independent of particle size throughout the series, to at least the 10 μm fraction. Further tests using different weights of tailings showed that the BET-measured area is directly proportional to the sample mass. Hence, the overall results indicate that the tailings are highly micro-porous, to the extent that screenings at particle sizes >10 μm are irrelevant in terms of the screen-size specifications for the NP tests.

The NP tests of the tailings using the standard volume and concentration of acid (20 mL, 0.1N) gave fairly constant NP values of 45-50 for all the particle sizes >10 μm (Table 3). Tests using larger volumes and higher acid concentrations substantially increased the NP values (Table 3), once again demonstrating the importance of testing the material at the appropriate acidity level [2, 4, 21] lest the NP be overestimated.

Table 3. Geometric versus BET surface areas for serpentine tailings

Mesh	μm	d_c μm	A_{geo} m^2/g	BET m^2/g	NP 0.1N, 20mL	NP 0.5N, 20mL	NP 0.5N, 80mL
–70+150	210-105	151.5	0.0155	9.22	45		398
–150+200	105-74	88.6	0.0266	10.01	49		380
–200+270	74-53	62.9	0.0374	10.63	50		449
–210+325	53-44	48.4	0.0487	10.39	50		455
–325+400	44-37	40.4	0.0582	10.62	50		442
cyclone	37-20	27.6	0.0852	8.73	50	311	396
cyclone	20-10	14.4	0.1632	10.67	50	337	404
cyclone	<10	4.34	0.5418	18.72			419

Conclusions

The Sobek tests of various minerals have shown that normalization of the NP to unit area may give misleading results in cases where the BET-measured surface area is low, thereby giving inordinately high normalized NP values. Although the NP increases as the particle size decreases, the change in NP is only a few units over the size range specified for most static tests. The method of grinding a sample may affect the NP, and high-impact methods, especially if applied to coarse-grained micaceous minerals, will likely increase the NP values because of the creation of more sites that are particularly susceptible to acid dissolution. A more important control on NP, however, is the amount of fines that is included in a test-sample; although the NP varies only slightly over the sizes specified for most static tests, it increases substantially once the particle size is reduced to less than about 50 μm. Static-test protocols currently specify only that the sample be screened to pass a particular mesh, with no requirement for a lower limit on particle size. Even though the NP of bulk samples increases by only a few units over the passing sizes commonly specified in various static-test methodologies, the trend clearly is toward higher NP values as the specified screen-passing size becomes finer.

Aside from the systematic variations between NP and particle size, a novel example of the effects of extreme microporosity is demonstrated for serpentine tailings. The BET-measured surface areas of the tailings do not change until the particle size decreases to about 10 μm; consequently, the NP values are unaffected over a wide range of screened particle sizes.

Acknowledgements

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